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(54) Title: **ANTIPERSPIRANTS**

(57) Abstract: Antiperspirant products comprise a dispensing container for a soft solid having a narrow aperture through which its contents can be dispensed. The contents comprise a particulate antiperspirant active suspended in a water-immiscible liquid which has a refractive index that differs from that of the antiperspirant active by preferably not more than 0.08. By controlling the liquid and solid phase refractive indexes to within such a difference, it is possible to dispense a ribbon of soft solid through the narrow aperture which has a translucent appearance, even if the gross formulation within the dispenser is opaque. Particularly desirably, the refractive indexes differ by at least 0.01 or 0.02 to 0.06, in order to additionally avoid formulation constraints that would apply in respect if the liquid and suspended active had to be exactly matched.

WO 01/58411 A2

- 1 -

ANTIPERSPIRANTSFIELD OF THE INVENTION

5 The present invention relates to soft solid antiperspirant compositions for application to human skin, especially the axilla.

BACKGROUND OF THE INVENTION AND SUMMARY OF PRIOR ART

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Antiperspirant compositions are widely used in order to enable their users to avoid or minimise wet patches on their skin, especially in axillary regions. A variety of these compositions make use of a thickened or structured liquid
15 which is applied to the surface of the skin and serves as a carrier for the antiperspirant active. In many such compositions the liquid is water-immiscible and is thickened or structured by one or more materials incorporated into the composition for that purpose, and
20 usually referred to as a "structurant" or "gellant".

Antiperspirant formulations have been provided with a range of different product forms. One of these is a so-called "stick" which is usually a bar of an apparently firm solid
25 material held within a dispensing container and which retains its structural integrity and shape whilst being applied. Another possibility is a softer solid composition accommodated in a dispensing container which in use extrudes the composition through one or more apertures.

30 The present invention is concerned with such soft solid compositions. Such compositions have sufficient rigidity

- 2 -

that they are not observed by the human eye to flow, but they are deformable by hand pressure and can be extruded from a container through one or more apertures at the end of the container.

5

For use a small amount of the composition is extruded from the container, which may then be used as an applicator to spread the extruded material on the skin.

- 10 A number of properties of such compositions are significant. The composition should be stable and not leak from its container until deliberately extruded. Its sensory feel when applied should, desirably, not be sticky. The applied film of the composition preferably is of a transparent or
15 translucent appearance rather than an opaque white. This property is referred to as low visible residue, and it is desirable in order that the deposit on the user's skin is not easily seen. Moreover, this also avoids conspicuous marks on clothing, to which the deposited material can
20 accidentally transfer.

There have been proposals to formulate other forms of antiperspirant product so as to be transparent. This is attractive to the consumer, but is difficult to achieve.

- 25 What is required is that the refractive indices of ingredients of the composition are closely matched, typically differing by less than 0.003. This is difficult to achieve and imposes constraints on the formulation which are detrimental to other properties perceived by the
30 consumer. Specifically, some transparent sticks which have been marketed were solidified solutions with a sticky feel

- 3 -

arising from a substantial content of strongly polar organic solvent.

It is also known to make transparent products which are
5 emulsions with closely matched refractive indices of the two
phases. They are commonly described as clear gels, and
frequently do not contain a structurant. They are perceived
by users as excessively wet and they tend to suffer a
localised loss of clarity after the package has been opened
10 and used, as a result of loss of volatiles from the unused
remainder, so that the unfinished product appears to have
deteriorated.

There have been suggestions in the literature that a
15 mismatch of refractive index as large as 0.02 will not
destroy transparency. We have found that compositions
according to this teaching do not appear transparent.

SUMMARY OF THE INVENTION

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We have now found that by some control of the formulation of
a soft solid which is a suspension of particulate
antiperspirant active together with an appropriate package
(which may be variations of a type currently in use for
25 opaque soft solid antiperspirant compositions) it is
possible to create a product where the dispensed material
has an attractively translucent appearance prior to
application onto the skin.

If the container has apertures for extrusion of composition,
30 which are of fairly small cross section, that is to say
sufficiently narrow in at least one dimension, the extruded

- 4 -

composition can have an attractively translucent appearance provided the refractive index of the liquid in the composition does not differ from that of the antiperspirant active by more than a limited amount, possibly about 0.10, and preferably not by more than about 0.08 or 0.07.

The translucent appearance provides an indication to the consumer that the deposit of the composition on skin (or accidental deposit on clothing) will be transparent or have low visibility.

There is no necessity to use antiperspirant active of exceptionally tiny (i.e. nanometre scale) particle size, (which would be another route to translucency).

15

Accordingly, in a first aspect, this invention provides an antiperspirant product comprising (i) a dispensing container provided with one or more outlet apertures for a user to expel material from the container through the said outlet aperture or apertures, and (ii) within said dispensing container, a soft solid antiperspirant composition comprising a continuous phase of water-immiscible liquid, at least one structuring material which thickens said liquid, and a particulate antiperspirant active in suspension in said liquid; wherein the refractive indices of said antiperspirant active and said liquid differ by not more than about 0.10 preferably not more than 0.08 or 0.07.

The water-immiscible liquid will generally be a mixture of liquids and the refractive index of that mixture should not differ by more than 0.10 and very preferably not more than

- 5 -

0.08 from that of the antiperspirant active or mixture of actives.

It is envisaged that the refractive indices will usually
5 differ by at least 0.01 or 0.02 - avoiding the more difficult constraints on the formulation needed to achieve a closer match. Preferably the difference will not exceed about 0.06.

10 The structurant materials may come from several categories. Their purpose is to increase the viscosity of the water-immiscible liquid and therefore the whole composition, so that in the container before extrusion at the time of use it is a soft solid.

15 A class of materials which have traditionally been used as structurants are fatty alcohols, which are solid at 20°C. These are not preferred for this invention because they give an opaque white appearance with a high visible residue.

20 One category of materials which may be used is waxes which are solid at temperatures up to at least 30°C yet generally melt at a temperature not over 95°C. An amount from 3% or 4% up to 8% by weight of the composition may be suitable.

25 Another category of structuring material is those which cause gelation by forming a network of fibres within the composition as it cools from a heated state during processing. An amount from 1% up to 6% by weight of the
30 composition may be suitable.

- 6 -

A further and preferred possibility is an organic polymeric thickener which is effective to increase the viscosity of the water-immiscible liquid. If used alone, the amount of such polymer is likely to be from 3% or better 5% up to 20%
5 by weight of the composition.

In accordance with a co-pending application another preferred structuring system is a combination of such an organic polymer together with second structuring material
10 selected from:

- a) structurant which forms a network of fibres within the continuous phase
- b) waxes, other than fatty alcohols, which are solid at
15 temperatures of 30°C and below, but melt below 95 C, and
- c) mixtures thereof.

In such a system the amount of polymeric thickener
20 preferably lies in a range from 1.5% to 15% by weight of the composition. If structurant (a) which forms a network of fibres within the continuous phase is present, the amount of it will generally be from 0.5 or 1% to 7% by weight of the composition. If wax (b) is present the amount of it will
25 generally be from 0.5 or 1% to 15% by weight of the composition. More preferably, the total amount of second structuring material may be from 1% to 7% by weight of the composition and the total of organic polymer and second structuring material may be from 3% or 4% up to 10% or 12%
30 by weight of the composition.

- 7 -

Among the various possibilities above, it is preferred to use a polymeric thickener, alone or in conjunction with a second structuring material. Whether or not the structurant includes polymer, fibre-forming structurants are sometimes preferred over those waxes which do not form fibres, and on other occasions the use of non-fibre-forming waxes is preferred.

In many instances the total amount of the organic polymeric thickener and second structuring material will be greater than the total amount of any fatty alcohol which is solid at 20 C. There is no necessity to incorporate any such fatty alcohol. Preferably such fatty alcohol is excluded or used only at low concentrations, such as not more than 3% by weight of the composition, better not more than 1.5% since it is known to crystallize as relatively large platelets and increase the opacity and visibility of deposits.

As mentioned, the container should have provision for a user to expel material through the outlet apertures. A container will generally have a body part dimensioned to be held in the hand and outlet aperture(s) at one end.

Means for urging the contents of the container to the said aperture or apertures, for flow through them, may be moving parts operable by the user or may simply be flexible container walls so that the user can expel composition from the container by squeezing it.

One form of container has a barrel or tube to hold a quantity of the composition and a component part referred to as an elevator or piston which is capable of movement within

- 8 -

the barrel or tube to propel the composition towards the outlet aperture(s).

Preferably then the container also includes a transport
5 mechanism for moving the piston comprising a threaded rod
which extends axially into the body of the composition
through a correspondingly threaded aperture in the piston,
and means for relatively rotating the rod and piston.
Conveniently this is a hand-operable mechanism or wheel
10 (such as a handwheel or pawl and ratchet) located at the
opposite end of the container to the delivery opening.

The outlet apertures are normally formed in a closure for
the container. The apertures should each be such that the
15 material extruded through it is no more than 0.5cm thick in
at least one dimension. Thus an aperture could have a cross
section with its smallest dimension less than 0.5cm wide. A
rectangle 0.4cm by 0.8cm would be an example. The material
extruded through it would be a ribbon 0.4cm thick.

20 Possibly each aperture will have a cross section which is
not more than 0.5cm in any dimension. A circular hole of
0.5cm diameter would be an example.

25 Subject to the foregoing requirements, the number and design
of such apertures is at the discretion of the designer of
the package. Likewise, the distribution of such apertures
in the dispensing head of the container is at the discretion
of the package designer.

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- 9 -

In practice, the apertures are often circular or oval or parallel sided slots, the slots being straight, curved or serpentine, often having rounded ends, or a combination of two or more designs. The number of apertures is often from 5 to 150, especially 8 to 120. The apertures are often arranged in a symmetrical manner in the dispensing head, such as parallel slots, or in a radial design centred on the container axis, or a rectangular array of circular or oval apertures, or in concentric or intersecting circles.

10

The component parts of such containers are often made from thermoplastic materials, for example polypropylene or polyethylene. Descriptions of suitable containers, some of which include further features, are found in US patents 4865231, 5000356 and 5573341 and WO 99/29585.

15

Compositions with the preferred structurants (i.e. polymer or a combination including polymer) are simple to manufacture. The compositions can be made and packed by heating their constituents to form a liquid composition, mixing at temperatures where the composition is freely mobile, putting the composition into containers for retail sale and cooling or allowing these compositions to cool to room temperature. There is then no need for continued stirring while the composition is thickening as it cools.

20

There is provided a method for preventing or reducing perspiration on human skin comprising topically applying to the skin a composition as specified in the first aspect of this invention.

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- 10 -

BRIEF DESCRIPTION OF THE DRAWINGS

Fig. 1 is a perspective view of a dispensing container.

Fig. 2 shows the body of the container, with the upper part
5 in section.

Fig. 3 is an enlarged top view showing the outlet openings.

Fig. 4 shows an alternative outlet opening.

Fig. 5 shows the three parts of a different dispensing
container.

10 Fig. 6 is an enlarged end view, in the direction of arrow VI
in Fig. 5, with the detachable cap removed to show the
outlet apertures.

Fig. 7 is a diagrammatic cross sectional view of a third
form of dispensing container, with its upper part in
15 section.

Fig. 8 is an enlarged top view of the container of Fig. 7.

- 11 -

DETAILED DESCRIPTION AND EMBODIMENTSDispensing Containers

5 A first example of a dispensing container is shown in Figs.
1 to 3 of the drawings. This container has detachable cap 3
which is removable and replaceable by the user. The
container has a barrel 5 of oval cross section. The upper
end of the barrel is secured by a snap-fit (indicated at 8
10 in Fig. 2) to a top portion 10.

Snap-fitted into an upper opening in the top portion 10 is
an insert 6 which defines outlet apertures 7. The lower end
of the barrel 5 is closed by a base-plate 12.

15 The dome-shaped cap 3 is transparent so that the openings 7
in the insert 6 are visible. The interior surface of the
cap 3 is provided with stops 4 serving to maintain a head
space between the cap 3 and the top portion 10. The base-
20 plate 12 provides a mounting for a knurled hand wheel 13
attached to a threaded spindle 14. This, as shown in Fig. 2
extends within the barrel 5 and engages a threaded central
aperture in an elevator piston 15 within the barrel 5.

25 By turning the hand wheel 13 with the fingers, the user can
raise the piston 15 within the barrel 5 to push composition,
contained in the barrel above the elevator piston 15 towards
the outlet apertures 7. The composition is then expelled
through the apertures 7. In this way the user can dispense
30 composition from the container through the outlet aperture 7
as required.

- 12 -

The outlet apertures 7 are curved slots each having a uniform width, as indicated for example between the arrows (A), which is approximately 2 mm. The composition is therefore expelled from the container as a plurality of
5 ribbons each about 2mm thick.

Before the container is filled, the elevator piston 15 is positioned at the bottom of the container and the insert 6 is not fitted, thus leaving a fairly large opening at the
10 top of the container through which molten composition can be poured in, on top of piston 15. After the container is filled, the insert 6 is snapped into the opening in the top portion 10, the cap 3 is fitted and the filled container is ready for retail sale. If desired an adhesive label may be
15 secured onto the insert 6 closing the apertures 7 until this adhesive label is torn off by the user prior to first use.

Fig. 4 shows a minor modification. Instead of the plurality of slots 7 in the insert 6, there is a single central hole
20 18 with a diameter of 4mm.

In a modification of Fig. 4, not shown, the single central hole is replaced by an array of between 30 and 130 holes, e.g. about 120 holes, each with a diameter between 1 and
25 3mm.

Fig. 5 and Fig. 6 show a second example of dispensing container which is described and illustrated in greater detail in PCT publication WO 99/29585.

- 13 -

This container has a main body 22 with a rounded, closed first end 24 and a second end 26. The second end 26 has an open mouth 28. The container also includes a closure 32 with an oval end face 34 and a surrounding skirt 36. When
5 the closure 32 is fitted to the end 26 of the main body 22, a short internal wall 38 on the closure snap-fits to the mouth 28 of the main container 22. A detachable cap 40 fits over the closure 32 and an end portion of the main body 22. This cap 40 can be removed and replaced by the user.

10

As shown in Fig. 6, the face 34 of the closure 32 has three outlet apertures 42 in a generally central position.

The main body 22 is relatively rigid in the region of its closed, rounded end 24 but a middle area of the walls of
15 this container body is relatively flexible to allow it to be squeezed by the user. When the container body is squeezed in this way its contents are propelled towards the mouth 28 and a portion is extruded through the outlet apertures 42. Each of these outlet apertures is a curving slot with a
20 width, indicated for example between the arrows (A), of approximately 1mm. Consequently when the user squeezes the container body 22, material from it is extruded as three ribbons each 1mm thick.

25 This container is intended to be stored resting on the removable cap 40 and with the container mouth 28 downwards. For this reason the container cap 40 incorporates plugs 46 to fit into, and thereby block, the outlet apertures 42.

30 The container of Figs. 5 and 6 is filled with molten composition by pouring that composition in through the mouth

- 14 -

28. After this the closure 32 is fitted onto the end 26 of the container body and, as mentioned, the internal wall 38 within the closure 32 snap-fits onto the mouth 28 of the container body 22, so that it is then held in place.

5

Figs. 7 and 8 diagrammatically illustrate a third example of dispensing container. This is currently in commercial use for opaque soft solid compositions. This container has a downwardly tapering main body 50 of generally oval, cross section onto which is snap-fitted a closure 52. Rotatably mounted within the main body 50 is a tube 54 of circular cross section integral with a user-operable hand wheel 56 exposed at the base of the body 50. A threaded spindle 60 is accommodated centrally within the tube 54. The top of this spindle 60 engages a socket 62 of the end cap 52 and is thereby prevented from rotating. An elevator piston 64 has a threaded central aperture engaging the thread of the spindle 60. The top cap 52 incorporates a depending skirt 66 encircling the top of the tube 54.

20

The top cap 52 has eight apertures 68 each of which is a slot with a width, as shown between arrows (A), of 3mm and a length of approximately 12mm. Adjoining one edge of each aperture 68 is a blade 70 which projects downwardly and is also somewhat inclined. As a result, if the container is empty, the blades 70 are visible through the apertures 68 as seen in Fig. 8.

These containers are filled with the piston 64 at its lowest position and before the closure 52 is fitted. Molten composition is poured into the central tube 54 on top of the

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- 15 -

elevator piston 64. The space between the tube 54 and the body 50 remains empty. The top closure 52 is then snap-fitted onto the body 50 and its socket 62 engages the top of spindle 60.

5

For use the user turns the hand wheel 56 and by doing so turns the central tube 54 and the composition within it relative to the body 50. A ratchet, not shown, constrains the direction of rotation. The elevator piston 64
10 frictionally engages the tube 54 and rotates along with it and with the composition immediately above it. However because the central spindle 60 does not rotate the elevator piston 64 moves up the spindle 60, thereby propelling the composition up towards the outlet apertures 68. As the user
15 turns the wheel 56, the inclined blades 70 scoop from the top of the composition in the tube 54 and hence assist in guiding the flow of composition up to and through the outlet apertures 68.

20 A removable and replaceable transparent cap, not shown, is also provided to be placed over the closure 52 and top portion of the body 50.

It should be understood that these three embodiments of
25 dispensing container have been illustrated and described by way of example only and serve to show diversity of details amongst dispensing containers which may be used with compositions to give products of the present invention.

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- 16 -

COMPOSITIONS

The various materials which may be present in a composition of this invention will now be discussed by turn and preferred features and possibilities will be indicated.

5

Water-immiscible liquid

The water-immiscible liquid comprises one or a mixture of materials which are relatively hydrophobic so as to be
10 immiscible in water. Some hydrophilic liquid may be included, provided the overall liquid mixture is immiscible with water. Generally, this liquid or liquid mixture (when in the absence of polymeric thickener or other structurant) will be freely mobile at temperatures of 15 C and above. It
15 may have some volatility but its vapour pressure will generally be less than 4kPa (30 mmHg) at 25°C so that the material can be referred to as an oil or mixture of oils. More specifically, it is desirable that at least 80% by weight of the liquid should consist of materials with a
20 vapour pressure not over this value of 4kPa at 25°C.

It is possible that the liquid or liquid mixture includes a volatile liquid silicone, i.e. liquid polyorganosiloxane. To class as "volatile" such material should have a
25 measurable vapour pressure at 20 or 25°C. Typically the vapour pressure of a volatile silicone lies in a range from 1 or 10 Pa to 2kPa at 25°C.

An advantage for including volatile silicone is that it
30 gives a "drier" feel to the applied film after the composition is applied to skin. However, for this invention

- 17 -

it is preferred to use it in a limited amount not exceeding 20% by weight of the composition because it has a low refractive index.

5 Volatile polyorganosiloxanes can be linear or cyclic or mixtures thereof. Preferred cyclic siloxanes include polydimethylsiloxanes and particularly those containing from 3 to 9 silicon atoms and preferably not more than 7 silicon atoms and most preferably from 4 to 6 silicon atoms,
10 otherwise often referred to as cyclomethicones. Preferred linear siloxanes include polydimethylsiloxanes containing from 3 to 9 silicon atoms. The volatile siloxanes normally by themselves exhibit viscosities of below 10^{-5} m²/sec (10 centistokes), and particularly above 10^{-7} m²/sec (0.1
15 centistokes), the linear siloxanes normally exhibiting a viscosity of below 5×10^{-6} m²/sec (5 centistokes). The volatile silicones can also comprise branched linear or cyclic siloxanes such as the aforementioned linear or cyclic siloxanes substituted by one or more pendant $-O-Si(CH_3)_3$
20 groups. Examples of commercially available silicone oils include oils having grade designations 344, 345, 244, 245 and 246 from Dow Corning Corporation; Silicone 7207 and Silicone 7158 from Union Carbide Corporation; and SF1202 from General Electric.

25

The hydrophobic liquid employed in compositions herein can alternatively or additionally comprise non-volatile silicone oils, which include polyalkyl siloxanes, polyalkylaryl siloxanes and polyethersiloxane copolymers. These can
30 suitably be selected from dimethicone and dimethicone

- 18 -

copolyols. Commercially available non-volatile silicone oils include Dow Corning 556 and Dow Corning 200 series.

Silicon-free hydrophobic liquids can be used instead of, or
5 more preferably in addition to liquid silicones. Silicon-free hydrophobic organic liquids which can be incorporated include liquid aliphatic hydrocarbons such as mineral oils or hydrogenated polyisobutene, often selected to exhibit a low viscosity. Further examples of liquid hydrocarbons are
10 polydecene and paraffins and isoparaffins of at least 10 carbon atoms. Although polyisobutene and polydecene are polymeric in nature, they are mobile liquids at room temperature of 20 C and do not cause thickening of other hydrophobic oils.

15 Some hydrophobic aliphatic or aromatic esters are liquids. Aromatic esters may be preferred and may well be used as all or part of a liquid mixture; aliphatic esters tend to have lower refractive index and for this reason they will
20 generally only be used as part of a liquid mixture.

Suitable aliphatic esters contain at least one long chain alkyl group, such as esters derived from C₁ to C₂₀ alkanols esterified with a C₈ to C₂₂ alkanolic acid or C₆ to C₁₀
25 alkanedioic acid. The alkanol and acid moieties or mixtures thereof are preferably selected such that they each have a melting point of below 20 C. These esters include isopropyl myristate, lauryl myristate, isopropyl palmitate, diisopropyl sebacate and diisopropyl adipate.

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- 19 -

Suitable liquid aromatic esters, preferably having a melting point of below 20 C, include fatty alkyl benzoates, alkylene dibenzoate, alkoxyated alkyl benzoate or a polyalkylene oxide dibenzoate, or a mixture of two or more. The alkyl group often contains at least 8 carbons, in many instances up to 25 carbons, eg from C8 to C18. It is often linear, but can alternatively be branched. Especially desirable alkyl groups are found in the range of from 12 to 20 carbons and include dodecyl (lauryl) terdecyl, tetradecyl (myristyl), pentadecyl, hexadecyl (palmityl), octadecyl (stearyl) 2-methyl-heptadecyl (iso-stearyl) and octyldodecyl groups. A mixture of two or more of the alkyl groups can be employed, such as a mixture of C12-C15 alkyl groups. The term alkylated herein includes alkylene groups and the latter are terminated at each end with a benzoate group. The alkylene group often contains from 2 to 6 carbons and can be linear or branched, a suitable example of linear being propylene.

In alkoxyated alkyl benzoate esters, the alkyl group is terminated by an alkoxy group, which can be monomeric containing for example up to 6 carbons or polymeric such as polyethylene oxide or preferably polypropylene oxide, which conveniently comprises up to 30 units and often from 5 to 20 units. In such compounds, the alkyl group can be selected from the previously identified alkyl groups. Alternatively, the benzoate compound can comprise a polyethylene oxide or polypropylene oxide moiety, or preferably a block copolymer of ethylene oxide and propylene oxide, terminated at each end by a benzoate group.

- 20 -

As mentioned above, aliphatic alcohols which are solid at 20 C, such as stearyl alcohol are preferably absent or else present in low concentration such as less than 5% by weight of the whole composition since these lead to visible white deposits when a composition is used.

However, aliphatic alcohols which are liquid at 20 C may be employed. These include branched chain alcohols of at least 10 carbon atoms such as isostearyl alcohol and octyl dodecanol.

Silicon-free liquids can constitute from 0-100% of the water-immiscible liquid, but it is preferred that some silicone oil is present while the amount of silicon-free constituents preferably constitutes at least 75% of the water-immiscible liquid.

Organic polymeric thickener

A number of organic polymers are effective to increase the viscosity of hydrophobic liquids, although some polymers do not do so.

A material which is suitable as an organic polymeric thickener will generally possess the following characteristics:-

- i) it will contain residues of at least 5 (possibly many more than 5) monomer units bonded together into a polymer chain

- 21 -

- ii) it should dissolve on heating in water-immiscible liquids, and specifically it must have a solubility of at least 1.5% by weight in the heated water-immiscible liquid of the continuous phase;
- 5 iii) after heating to dissolve and cooling to 20 C, it will increase the viscosity of the water-immiscible liquid of the continuous phase, in the absence of other structurant, when dissolved therein at the same concentration as in the formulation of the
10 invention.

Preferably, under these conditions, it will bring about a viscosity increase of at least 100 mPa.sec, better at least 250 mPa.sec when viscosity is measured with a Brookfield
15 viscometer using a T-bar spindle at 10 rpm at 20°C. The choice of a type B, type C or type D T-bar spindle will depend on the viscosity of the system being measured. Provided the spindle is appropriate to provide a viscosity measurement it will enable determination of an increase in
20 viscosity brought about by the polymer.

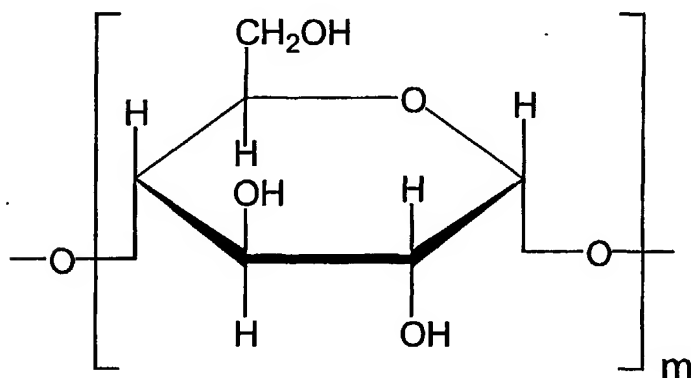
An additional or alternative characterisation of a suitable polymer is that it can thicken the water-immiscible liquid to a viscosity of at least 10,000 mPa.sec, measured in the
25 same way, when incorporated in the water-immiscible liquid at 15% by weight, in the absence of the other structurant.

The polymer will generally be solid at 20°C.

- 22 -

One category of polymer which has been found suitable is a polysaccharide esterified with monocarboxylic acid containing at least 4 carbon atoms.

- 5 Preferred in this category is a dextrin fatty acid ester having the formula:



- 10 wherein each R group, individually, is a hydrogen atom or an acyl group having up to 22 carbon atoms, provided that at least one R group per glucose unit is an acyl group of at least 4 carbon atoms, and m has an average value from 5, 10 or 20 up to 50 or even up to 150, more preferably from 20 to 30. The dextrin fatty acid ester can be a partial ester, i.e. at least one R group is hydrogen; or the dextrin can be completely esterified, i.e., all R groups are acyl, such as a C₄ - C₂₂ acyl group. The acyl groups may be the same or similar, and preferably they are straight chain acyl groups with chain lengths of 8 to 22 carbon atoms, e.g. in a range from 12 or 14 carbon atoms to 18 or 20 carbon atoms. Branched acyl groups may be included, possibly as in a mixture of C₆ to C₂₂ linear acyl groups. Shorter acyl groups may form part of a mixture, for example C₄ to C₈ acyl groups

- 23 -

may be mixed with C_{12} to C_{22} linear acyl groups. In preferred embodiments, wherein the R group is a C_8 - C_{22} acyl group the degree of substitution is at least 2 (i.e., at least two R groups are C_8 - C_{22} acyl groups).

5

The C_8 - C_{22} fatty acids that are reacted with the starch hydrolyzate can be saturated or unsaturated acids, and include, for example, capric acid, pelargonic acid, caprylic acid, undecylic acid, undecylenic acid, lauric acid, myristic acid, pentadecylic acid, palmitic acid, heptadecylic acid, stearic acid, nonadecanoic acid, arachic acid, oleic acid, linoleic acid, linolenic acid, similar acids, and mixtures thereof. These dextrin fatty acid esters are disclosed in Mori et al US patent 4780145, incorporated herein by reference, and some of them are available under the trade name RHEOPEARL from Chiba Flour Milling Co., Ltd., Japan. An example of a dextrin fatty acid ester is dextrin palmitate, available commercially as RHEOPEARL KL and RHEOPEARL FL, for example, from Chiba Flour Milling Co., Ltd. Other examples of esters of C_8 - C_{22} carboxylic acids are dextrin behenate, dextrin laurate, dextrin myristate, dextrin stearate, and mixtures thereof.

10
15
20

A second category of polymer which can be used as a thickener is polyamides as discussed in US 5500209. Such polyamides may be derived from organic diamines containing 2 to 12, preferably 2 to 8 carbon atoms, condensed with di- or poly carboxylic acids containing 4 to 20 carbon atoms per carboxylic acid group. Some monocarboxylic acid may be included in the reaction mixture to control polymer chain

25
30

- 24 -

length. The dicarboxylic acids may be obtained by thermal polymerisation of unsaturated monocarboxylic acids.

Such polyamides are available from Henkel under their trade
5 name VERSAMID. An example is VERSAMID 950 from hexamethylene diamine and adipic acid.

A further category of polymer which has been found useful is the block copolymers of styrene with ethylene propylene and/or butylene available from Shell under their trade name
10 KRATON G.

Preferred in this category is styrene ethylene/butylene styrene linear block copolymers e.g. that available as KRATON G 1726X.

15 Another suitable type of polymer is polymers of alpha methylstyrene and styrene available from Hercules under the trade name KRISTALEX. One suitable grade is KRISTALEX F85, with mean molecular weight of approximately 1200.

20 Yet another polymer found to be suitable although less preferred is alkyl substituted galactomannan available from Hercules under their trade name N-HANCE AG.

25 A still further class of polymers found to be suitable comprises co-polymers of vinyl pyrrolidone with polyethylene containing at least 25 methylene units. A particularly suitable polymer comprises triacontanyl polyvinylpyrrolidone, such as that available from
30 International Speciality Products under the trade name Antaron WP-660.

- 25 -

The thickening ability of polymers varies from one to another, which will affect the amount which is required. The amount will often lie in a range from 2% or 3% by weight of the composition up to 7% or more, such as to 10%, 12% or
5 15%.

Fibre-forming Structurant

A number of organic compounds are known to possess the ability to gel hydrophobic organic liquids such as water-
10 immiscible hydrocarbon and/or silicone oils. Such materials are generally monomers or dimers with molecular weight below 10,000 often below 5,000 or even 1,000 rather than polymers with more than four repeat units or with molecular weight above 10,000.

15

Gel formation takes place as an exothermic event within a temperature range referred to as the gel point; upon reheating, melting of the gel takes place as an endothermic event within a temperature range. Such gels can be
20 disrupted by shearing. Although a small partial recovery may then be observed, such gels do not recover their structure for a long time, if at all, unless remelted.

Materials with this ability to gel hydrophobic organic
25 liquids have been reviewed by Terech and Weiss in "Low Molecular Mass Gelators of Organic Liquids and the Properties of their Gels" Chem. Rev 97, 3133-3159 [1997] and by Terech in Chapter 8, "Low-molecular weight Organogelators" of the book "Specialist surfactants" edited
30 by I D Robb, Blackie Academic Professional, 1997.

- 26 -

It is characteristic of such structurants, useful in this invention, that

- " they are able to gel the organic liquid in the absence of any disperse phase, when used in
5 sufficient quantity not exceeding 15% by weight;
- " the structured liquids are obtainable by cooling from an elevated temperature at which the structurant is in solution in the liquid - this hot solution being mobile and pourable;
- 10 " the (thus obtained) structured liquid becomes more mobile if subjected to shear or stress;
- " the structure does not spontaneously recover within 24 hours if the sheared liquid is left to stand at ambient laboratory temperature, even though a small
15 partial recovery may be observed;
- " the structure can be recovered by reheating to a temperature at which the structurant is in solution in the liquid and allowing it to cool back to ambient laboratory temperature.

20

It appears that such structurants operate by interactions which are permanent unless disrupted by shear or heating. Such structurants form a network of strands or fibres extending throughout the gelled liquid. In some cases these
25 fibres can be observed by electron microscopy, although in other cases the observation of the fibres which are believed to be present is prevented by practical difficulties in preparing a suitable specimen. When observed, the primary fibres in a gel are generally thin (diameter less than
30 0.5 μ m, often less than 0.2 μ m) and appear to have numerous

- 27 -

branches or interconnections. Primary fibres may entwine to form a thicker strand.

If these fibres are crystalline, they may or may not be the same polymorph as macroscopic crystals obtained by
5 conventional crystallization from a solvent.

One material which is well known to form such gels is 12-hydroxy stearic acid which is discussed in Terech et al "Organogels and Aerogels of Racemic and Chiral 12-hydroxy
10 octadecanoic Acid", Langmuir Vol 10, 3406-3418, 1994. The material is commercially available from Ajinomoto and also from Caschem.

US-A-5750096 is one of several documents which teaches that
15 gelation can be brought about using esters or amides of 12-hydroxy stearic acid. The alcohol used to form such an ester or the amine used to form such an amide may contain an aliphatic, cycloaliphatic or aromatic group with up to 22 carbons therein. If the group is aliphatic it preferably
20 contains at least three carbon atoms. A cycloaliphatic group preferably contains at least five carbon atoms and may be a fixed ring system such as adamantyl.

Other fatty acids with C₈ or longer alkyl chains may be used
25 and amides thereof can also be used. A specific example is lauric monoethanolamide also termed MEA lauramide.

N-acyl amino acid amides and esters are also known to structure liquids. We have established that they do so by
30 forming fibrous networks. They are described in US patent 3969087. N-Lauroyl-L-glutamic acid di-n-butylamide is

- 28 -

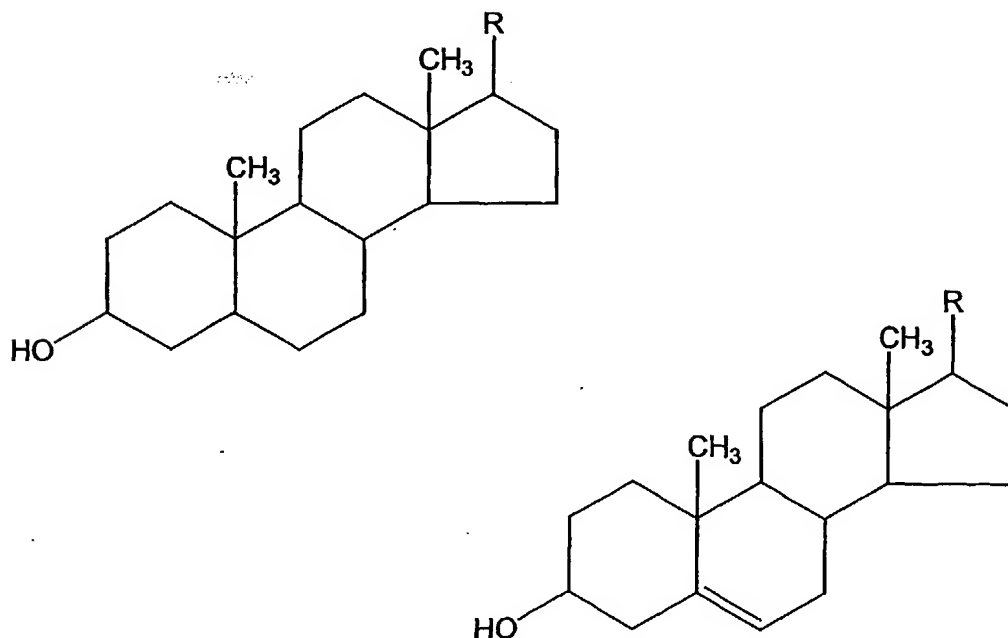
commercially available from Ajinomoto under their designation GP-1.

Further materials which have been disclosed as gelling
5 agents are the amide derivatives of di and tribasic
carboxylic acids set forth in WO 98/27954 notably alkyl
N,N'dialkyl succinamides.

A structurant which is the subject of a co-pending
10 application, published as WO 00/61096, is a combination of a
sterol and a sterol ester.

In its preferred form the sterol satisfies either of the two
formulae:

15



in which R represents an aliphatic, cycloaliphatic or
aromatic group, and preferably a linear or branched

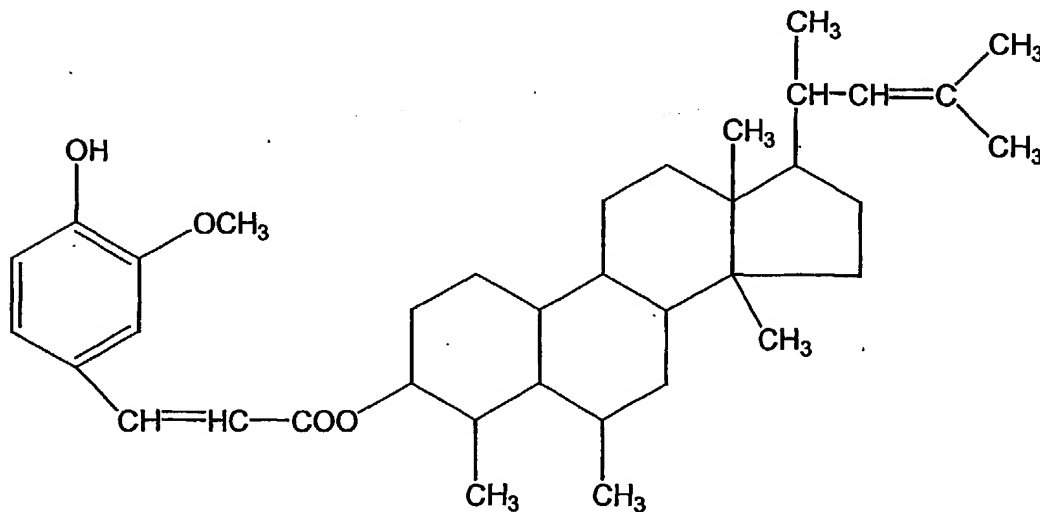
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aliphatic saturated or unsaturated hydrocarbon group. R desirably contains from 1 to 20 carbons and preferably from 4 to 14 carbons.

- 5 It is particularly suitable to employ β -sitosterol or campesterol or cholesterol, or a hydrogenated derivative thereof, such as dihydrocholesterol, or a mixture of two or more of them. An especially preferred sterol is β -sitosterol.

10

The preferred sterol ester is oryzanol, sometimes referred to as γ -oryzanol which contains material satisfying the following formula:-



15

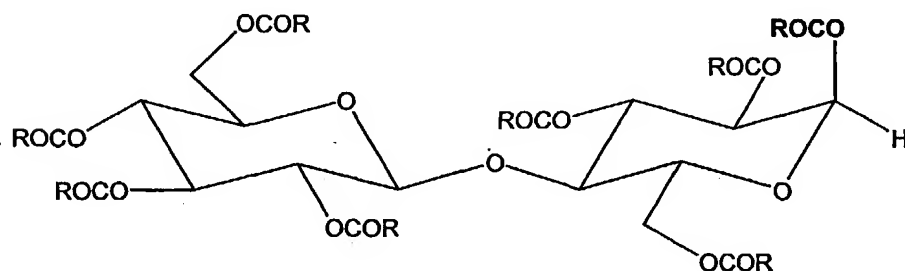
- The sterol and sterol ester are used in a mole ratio that is normally selected in the range of from 10:1 to 1:10, especially from 6:1 to 1:4 and preferably in the range of from 3:1 to 1:2. Employment of the two system constituents within such a mole ratio range, and especially within the
- 20

- 30 -

preferred range facilitates the co-stacking of the constituents and consequently facilitates the formation of a network that is readily able to structure the formulation.

5 Another structurant which is the subject of a co-pending application published as WO 00/61079 and which may be used in this invention is an ester of cellobiose and a fatty acid, preferably of 6 to 13 carbon atoms especially 8 to 10 carbon atoms. Preferably the cellobiose is fully
10 esterified, or nearly so, and is in the α -anomeric form.

The structure of such a compound, in its α -anomeric form is:



15

where R is an alkyl or alkenyl chain of 5 to 12 carbon atoms so that the acyl group contains 6 to 13 carbon atoms. Particularly preferred acyl groups incorporate a linear alkyl chain of 7 to 9 carbon atoms and are thus octanoyl,
20 nonanoyl or decanoyl.

The acyl groups may have a mixture of chain lengths but it is preferred that they are similar in size and structure. Thus it is preferred that all of the acyl groups are
25 aliphatic and at least 90% of the acyl groups have a chain length within a range such that the shorter and longer chain

- 31 -

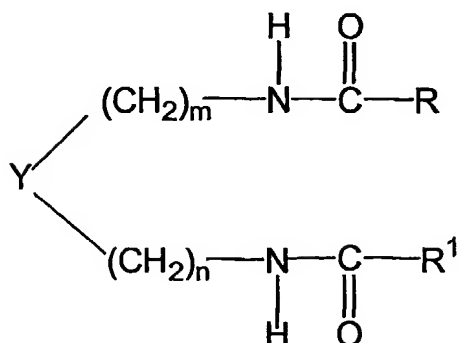
lengths in the range differ by no more than two carbon atoms, i.e. length in a range from $m - 1$ to $m + 1$ carbon atoms where m has a value in a range from 7 to 10.

- 5 Linear aliphatic acyl groups may be obtained from natural sources, in which case the number of carbon atoms in the acyl group is likely to be an even number or may be derived synthetically from petroleum as the raw material in which case both odd and even numbered chain lengths are available.

10

Synthetic methods for the esterification of saccharides are well known. The esterification of cellobiose has been reported by Takada et al in *Liquid Crystals*, (1995) Volume 19, pages 441-448. This article gives a procedure for the
 15 production of the alpha anomers of cellobiose octa-alkanoates by esterification of β -cellobiose using an alkanoic acid together with trifluoroacetic anhydride.

A further class of fibre-forming structurants which are
 20 described in a co-pending application, satisfy the general formula:-



- 32 -

in which R and R' are each independently a linear or branched moiety containing 5 to 27 carbons, m and n are each independently 0 or 1, and Y is a cyclohexane ring bearing the two amido substituents shown above meta or preferably
5 ortho to each other. Preferably R and R' are selected from C11 to C17. In the formula, m and n are preferably 1 when the amido substituents are meta to each other, and preferably 0 when the substituents are ortho to each other.

10 Waxes

This term "wax" is conventionally applied to a variety of materials and mixtures which have similar physical properties, namely that:-

- 15 they are solid at 30 C and preferably also at 40 C;
 they melt to a mobile liquid at a temperature above 30 C but generally below 95 C and preferably in a temperature range of 40 C to 90 C;
 they are water-insoluble and remain water-immiscible
20 when heated above their melting point.

Waxes are usually hydrocarbons, silicone polymers, esters of fatty acids or mixtures containing such compounds along with a minority (less than 50%) of other compounds. Naturally
25 occurring waxes are often mixtures of compounds which include a substantial proportion likely to be a majority of fatty esters.

Waxes form crystals in the water-immiscible liquid when it
30 cools from the heated state during processing.

- 33 -

These crystals take various forms including needles and platelets depending on the individual waxes. Some waxes form a network of fibrous crystals and can therefore also be identified as fibre-forming structurants.

5

Examples of hydrocarbon waxes include paraffin wax, microcrystalline wax and polyethylenes with molecular weight of 2,000 to 10,000.

- 10 Examples of ester waxes include esters of C_{16} - C_{22} fatty acids with glycerol or ethylene glycol and these may be made synthetically.

- Examples of natural waxes include beeswax, carnauba and
15 candelilla waxes which are of vegetable origin and mineral waxes from fossil remains other than petroleum. Montan wax, which is an example of mineral wax, includes non-glyceride esters of carboxylic acids, hydrocarbons and other constituents.

20

Further waxes employable herein comprise silicone polymer waxes, including waxes which satisfy the empirical formula:-
 $R-(SiMe_2-O)_x-SiMe_2R$

- in which x is at least 10, preferably 10 to 50 and R
25 represents an alkyl group containing at least 20 carbons, preferably 25 to 40 carbons, and particularly having an average linear chain length of at least 30 carbons.

- Other silicone waxes comprise copolymers of dimethicone and
30 alkyloxymethicone, satisfying the general formula:-
 $Y-(SiMe_2-O)_Y(Si[OR']Me-O)_Z-Y'$

- 34 -

in which Y represents $\text{SiMe}_2\text{-O}$, Y' SiMe_2 , R' an alkyl of at least 15 carbons preferably 18 to 22 such as stearyl, y and z are both integers, totalling preferably from 10 to 50.

- 5 Waxes useful in the present invention will generally be those found to thicken water-immiscible oils such as cyclomethicones when dissolved therein (by heating and cooling) at a concentration of 5 to 15% by weight.
- 10 If a wax is used which forms a network of fibres, the amount of it may be from 0.5 to 7% by weight of the composition. If a wax is used which does not form such a network, for instance a wax which crystallizes as spherulitic needles or as small platelets, the amount may well be from 2% or 3% up to 10%, 12% or 15% of the composition. Silicone waxes are an example of waxes which crystallize as small platelets.

The total amount of second structurant may range from 0.5% or 1% of the composition up to 9%, 10% or 15%.

20

The ratio of polymer to second structurant can vary considerably but in many instances it will lie in a range from 6:1 to 1:4.

- 25 In a number of embodiments the composition will contain 0.5 to 10% or 15% polymeric thickener, 0.5 to 7% of fibre-forming structurant and 2% to 10% of a wax such as silicone wax which does not crystallize as a network of fibres, all these percentages being by weight of the composition.

30

- 35 -

In other embodiments the composition contains from 5 to 15% by weight and preferably 8 to 12% by weight of a mixture of an organic wax and a silicone wax, particularly in a weight ratio of from 5:1 to 2:1.

5

Antiperspirant Actives

The composition will contain a particulate antiperspirant active. Antiperspirant actives are preferably incorporated
10 in an amount of from 0.5-60%, particularly from 5 to 30% or 40% and especially from 5 or 10% to 30 or 35% of the weight of the composition.

Antiperspirant actives for use herein are often selected
15 from astringent active salts, including in particular aluminium, zirconium and mixed aluminium/zirconium salts, including both inorganic salts, salts with organic anions and complexes. Preferred astringent salts include aluminium, zirconium and aluminium/zirconium halides and halohydrate
20 salts, such as chlorohydrates.

Aluminium halohydrates are usually defined by the general formula $Al_2(OH)_xQ_y \cdot wH_2O$ in which Q represents chlorine, bromine or iodine, x is variable from 2 to 5 and $x + y = 6$
25 while wH_2O represents a variable amount of hydration. Especially effective aluminium halohydrate salts, known as activated aluminium chlorohydrates, are described in EP-A-6739 (Unilever NV et al), the contents of which specification is incorporated herein by reference. In some
30 highly desirable embodiments of the present invention, the antiperspirant active is made by an improvement to the

- 36 -

process described in EP-A-6739. The improved process maintains of aluminium in dilute concentration during the formation of aluminium chlorohydrate species, and closely controls the ageing stage, in particular ageing at an elevated temperature and for a suitable ageing period in inverse relationship to each other.

Zirconium actives can usually be represented by the empirical general formula: $\text{ZrO}(\text{OH})_{2n-nz}\text{B}_z \cdot w\text{H}_2\text{O}$ in which z is a variable in the range of from 0.9 to 2.0 so that the value $2n-nz$ is zero or positive, n is the valency of B, and B is selected from the group consisting of chloride, other halide, sulphamate, sulphate and mixtures thereof. Possible hydration to a variable extent is represented by $w\text{H}_2\text{O}$. Preferable is that B represents chloride and the variable z lies in the range from 1.5 to 1.87. In practice, such zirconium salts are usually not employed by themselves, but as a component of a combined aluminium and zirconium-based antiperspirant.

The above aluminium and zirconium salts may have coordinated and/or bound water in various quantities and/or may be present as polymeric species, mixtures or complexes. In particular, zirconium hydroxy salts often represent a range of salts having various amounts of the hydroxy group. Zirconium aluminium chlorohydrate may be particularly preferred.

Antiperspirant complexes based on the above-mentioned astringent aluminium and/or zirconium salts can be employed. The complex often employs a compound with a carboxylate group, and advantageously this is an amino acid. Examples

- 37 -

of suitable amino acids include dl-tryptophan, dl-phenylalanine, dl-valine, dl-methionine and -alanine, and preferably glycine which has the formula $\text{CH}_2(\text{NH}_2)\text{COOH}$.

5 It is highly desirable in some formulations to employ complexes of a combination of aluminium halohydrates and zirconium chlorohydrates together with amino acids such as glycine, which are disclosed in US-A-3792068 (Luedders et al). Certain of those Al/Zr complexes are commonly called
10 ZAG in the literature. ZAG actives generally contain aluminium, zirconium and chloride with an Al/Zr ratio in a range from 2 to 10, especially 2 to 6, an Al/Cl ratio from 2.1 to 0.9 and a variable amount of glycine. Actives of this preferred type are available from Westwood, from Summit
15 and from Reheis.

The water content of hydratable aluminium or aluminium/zirconium antiperspirant active materials can be controlled to vary the properties of the material, such as
20 by controlling the conditions under which the material is recovered from its preparative mixture and dried, and/or by post manufacture contact with a selected amount of water. In many actives, the proportion of water in the active will be chosen within the range of from 6 to 18% by weight, and
25 sometimes advantageously from 11 to 18% by weight.

Other actives which may be utilised include astringent titanium salts, for example those described in GB 2299506A.

30 The proportion of solid antiperspirant salt in a composition normally includes the weight of any water of hydration and any complexing agent that may also be present in the solid active.

- 38 -

The particle size of the antiperspirant salts often falls within the range of 0.1 to 200 μ m with a mean particle size often from 3 to 20 μ m. Both larger and smaller mean particle sizes can also be contemplated such as from 20 to 50 μ m or
5 0.1 to 1 μ m. Antiperspirant actives which have substantial internal voids are preferably avoided because they do not have uniform refractive index. Such actives can be made more useful for this invention by milling. Various milling techniques can be employed, such as ball or swing milling.

10

Refractive Index Values

Particulate antiperspirant actives often have a refractive index substantially above 1.50, e.g. about 1.53 to about
15 1.56. That value can be brought down to somewhat lower value by hydration but we have found that it is not easy to obtain an antiperspirant active with a refractive index of 1.48 or below even if the active is partially hydrated to lower its refractive index.

20

The refractive index of a solid antiperspirant active can be determined by dispersing it in a number of oils or oil mixtures of differing refractive index. When the resulting dispersion is transparent, the refractive index of the oil
25 or oil mixture (which can be determined by conventional measurement) is a good approximation to the refractive index of the dispersed antiperspirant active.

Some examples of oils which may be used to make mixtures
30 which vary in refractive index and used for the purpose of such measurement are:

- 39 -

- volatile silicone (refractive index about 1.40)
C₁₂₋₁₅ alkyl benzoate (refractive index about 1.48)
which is available as Finsolv TN and/or
octylmethoxycinnamate (refractive index about 1.54)
5 which is available as Parsol MCX
Polyphenylsiloxane (DC710) (refractive index about
1.53).
Cinnamic aldehyde (refractive index about 1.62).
- 10 Providing a water-immiscible liquid with a refractive index
no more than 0.10 units away from that of the antiperspirant
active generally involves choosing an oil or oil mixture
with a refractive index no more than 0.10 units, preferably
no more than 0.08 units below that of the antiperspirant
15 active. For this reason it may be preferred that the water-
immiscible liquid has a refractive index of at least 1.43,
more preferably at least 1.46.

For the continuous phase, silicon-free water-immiscible
20 liquid oils generally have refractive indices in a range
from 1.43 to 1.49 at 22 C and can be used alone or mixed
together to give a silicon-free carrier liquid with
refractive index in this range. Volatile silicone oils
generally have a refractive index slightly below 1.40 at
25 22 C, but carrier liquid mixtures with refractive indices in
the range from 1.41 to 1.49 can be obtained by mixing
limited amounts of volatile silicone with other oils.
Cosmetically acceptable non-volatile silicone oils generally
have refractive indices in a range from 1.45 to 1.48 at 22 C
30 and so can be included when desired.

- 40 -

It may be desirable that the structurant materials also have refractive index differing from those of the water-immiscible liquid and the antiperspirant active by not more than 0.10 or 0.08 unit, although this is less critical. A number of structurant materials which can be used have refractive indices above 1.45. The refractive index of a wax or that of a fibrous network of a fibre-forming structurant can be determined (somewhat analogously to the determination for an antiperspirant active) by using that structurant to gel a number of oils or oil mixtures of differing refractive index. When the resulting gel is transparent, the refractive index of the oil or oil mixture is a good approximation to the refractive index of the structurant. The oils or mixtures of oils should be chosen from those which are gelled well by the structurant to avoid interfering effects.

Using this method we have determined the refractive indices of some structurants, namely:

N-lauroyl L-glutamic acid di-n-butylamide	approx 1.48
12-hydroxystearic acid	approx 1.52
α -cellobiose octa-esters with C ₈ to C ₁₂ fatty acids	approx 1.48

It appears that polymeric thickener causes very little scattering of light and its does not generally need to be taken into account for refractive index matching.

Optional Ingredients

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- 41 -

Optional ingredients in compositions of this invention can include deodorants, for example at a concentration of up to about 10% w/w. Suitable deodorant actives can comprise deoperfumes, and/or microbicides, including particularly bactericides, such as chlorinated aromatics, including biguanide derivatives, of which materials known as Triclosan (Igasan DP300™), Tricloban™, and Chlorhexidine warrant specific mention. A yet another class comprises biguanide salts such as available under the trade mark Cosmosil™.

Other optional ingredients include wash-off agents, often present in an amount of up to 10% w/w to assist in the removal of the formulation from skin or clothing. Such wash-off agents are typically nonionic surfactants such as esters or ethers containing a C₈ to C₂₂ alkyl moiety and a hydrophilic moiety which can comprise a polyoxyalkylene group (POE or POP) and/or a polyol.

The composition herein can incorporate one or more cosmetic adjuncts conventionally envisaged for antiperspirant soft solids. Such cosmetic adjuncts can include skin benefit agents such as glycerol, allantoin or lipids, for example in an amount of up to 5%; and soluble colourants. Skin cooling agents such as menthol and menthol derivatives, often in an amount of up to 2%, all of these percentages being by weight of the composition. A commonly employed adjunct is a perfume, which is normally present at a concentration of from 0 to 4% and in many formulations from 0.25 to 2% by weight of the composition.

- 42 -

The formulations can also include, if desired, a small particle sized inorganic thickener, often in an amount of from 0.1 to 2% by weight. Finely particulate silica, eg fumed silica, such as available as Aerosil™ 200 represents a preferred inorganic thickener.

Preparation

Compositions of this invention can be produced by conventional processes for making suspension solids or soft-solids. Such processes involve forming a heated mixture of the composition at a temperature which is sufficiently elevated that all the structurant dissolves, introducing that mixture into a mould, which may be a dispensing container, and then allowing the mixture to cool. If necessary, especially if the structurant does not include organic polymer, the composition may be subjected to shear mixing before it is put into the mould.

A convenient process sequence for a composition which is a suspension comprises first forming a solution of the polymer and other structurant in the water-immiscible liquid or liquid mixture. This is normally carried out by agitating the mixture at a temperature sufficiently high that all the structurant dissolves (the dissolution temperature) such as a temperature in a range from 50 to 150°C. Thereafter the particulate constituent, for example particulate antiperspirant active, is blended with the hot mixture. This must be done slowly, or the particulate solid must be preheated, in order to avoid premature gelation. The resulting blend is then introduced into a dispensing

- 43 -

container such as a stick barrel. This is usually carried out at a temperature 5 to 30°C above the setting temperature of the composition. The container and contents are then cooled to ambient temperature. Cooling may be brought about
5 by nothing more than allowing the container and contents to cool. Cooling may be assisted by blowing ambient or even refrigerated air over the containers and their contents.

It is highly desirable to control the manufacturing process
10 so as to avoid excessive entrapment of gas in the formulation, commonly in the form of small bubbles, and their retention in the packaged formulation. By so doing, homogeneity of the formulation is improved and clarity impairment of the formulation is lessened or even avoided.
15 The extent of gas entrapment can be avoided or at least kept to a minimum by suitably controlling the rate and manner of mixing the formulation whilst it is in the liquid or liquified state, and/or by employing a partial vacuum above the formulation, in accordance with known practice.
20 Likewise, retention of bubbles in formulation after filling into containers can be minimised or avoided by suitable control of the filling process, such as by locating a filling outlet in the agitation or storage vessel for the formulation remote from where bubbles tend to migrate, ie
25 remote from the upper volume of the formulation.

Measurement of Properties

i) Texture analyser

This test apparatus can move a blunt probe into or out from
30 a sample at a controlled speed and at the same time measure the applied force. The parameter which is determined as

- 44 -

hardness is a function of the force and the projected area of indentation.

A specific test protocol used a Stable Micro systems TA.XT2i
5 Texture Analyser. A sample of composition was made by heating the ingredients, pouring into a container and allowing to cool as described above. The container was a 15ml glass jar with a wide mouth. A metal sphere, of diameter 9.5mm, was attached to the underside of the Texture
10 Analyser's 5 kg load cell such that it could be used for indenting a sample placed beneath it on the base plate of the instrument. After positioning the sample, the sphere position was adjusted until it was just above the sample surface. *Texture Expert Exceed™* software was used to
15 generate the subsequent motion profile used in the test method. This profile initially moved the sphere into contact with the sample and then indented the sphere into the sample at an indentation speed of 0.05mm/s for a distance of 7mm. At this distance the direction of motion
20 of the sphere was immediately reversed to withdraw the sphere from the sample at the same speed of 0.05mm/s. During the course of the test, the data acquired were time(s), distance (mm) and force (N) and the data acquisition rate was 25 Hz.

25

The data associated with each test were manipulated using standard spreadsheet software and used to calculate the hardness, H at a travelled distance of 4.76mm after initial contact with the sample, using the following equation:

30

$$H = F/A$$

(H expressed in N.mm^{-2} , F in N and A in mm^2)

- 45 -

where F is the load at the same travelled distance and A is the projected area of the indentation. This area can be calculated geometrically and is equal to the area of a diametral plane of the sphere, i.e. $\pi (4.76)^2 \text{ mm}^2$.

5

For a soft solid composition the measured hardness will generally be from 0.003 to 0.5 Newton/mm². Frequently the hardness will be from 0.003 up to 0.1 Newton/mm².

10 ii) Whiteness of deposit

Another test of the properties of a composition is the whiteness and hence opacity of the composition which is delivered onto a surface when the composition is drawn across that surface. To carry out this test of deposition,
15 a sample of the composition was first applied to a test fabric under standardised conditions.

The test fabric was a rectangular strip of black worsted wool fabric 9cm by 15cm. This was placed in an apparatus
20 consisting of a metallic base onto which was hinged a metallic frame defining a rectangular aperture of 5cm by 9cm. The test portion of fabric was laid on the base. The hinged frame was placed over the fabric and secured to the base by means of two screws thereby clamping the test fabric
25 in place but exposing an area of 5 x 9 cm through the aperture.

A sample of soft solid composition in a dispensing container was kept at ambient laboratory temperature (about 20 C)
30 before it was required for measurement. A portion of the composition is then extruded from the container through the

- 46 -

dispensing apertures at one end. A weighed amount (0.5g) of the extruded composition was spread uniformly across the 5 x 9 cm area of test fabric enclosed by the frame. Spreading was carried out using a plastic spreading tool. After
5 spreading the sample of composition on the fabric substrate, it was removed from the apparatus and weighed to check that the mass of applied sample was 0.5 ± 0.01 gms.

The fabric with applied sample of composition was then
10 assessed twice for whiteness, once after one hour and again after 24 hours.

This measurement was carried out using a Sony XC77 monochrome video camera with a Cosmimar 16mm focal length
15 lens positioned vertically above a black table illuminated from a high angle using fluorescent tubes to remove shadowing. The apparatus was initially calibrated using a reference white card, after the fluorescent tubes had been turned on for long enough to give a steady light output.
20 The cloth with a deposit thereon was placed on the table and the camera was used to capture an image. An area of the image of the deposit was selected and analysed using a Kontron IBAS image analyser. This notionally divided the image into a large array of pixels and measured the grey
25 level of each pixel on a scale of 0 (black) to 255 (white). The average of the grey intensity was calculated. This was a measure of the whiteness of the deposit, with higher numbers indicating a whiter deposit. It was assumed that low numbers show a clear deposit allowing the substrate
30 colour to be seen. All samples were prepared in triplicate and a mean of the three measured values was reported.

- 47 -

iii) Light Transmission

The translucency of a composition may be measured by placing a sample of standardised thickness in the light path of a spectrophotometer and measuring transmittance, as a percentage of light transmitted in the absence of the gel.

We have carried out this test using a dual-beam spectrophotometer. The sample of composition was poured hot into a 4.5 ml cuvette made of polymethylmethacrylate (PMMA) and allowed to cool to an ambient temperature of 20-25°C. Such a cuvette gives a 1 cm thickness of composition. Measurement was carried out at 580 nm, with an identical but empty cuvette in the reference beam of the spectrophotometer, after the sample in the cuvette had been held for 24 hours. We have observed that a composition which gives a transmittance of only a fraction of 1% in this test is perceived by eye as "translucent" when extruded in a thickness less than 0.5cm. A transmittance measured at any temperature in the range from 20-25°C is usually adequately accurate, but measurement is made at 22°C if more precision is required.

The above test procedures were applied to two soft solid compositions currently marketed commercially, neither of which is translucent when stored within the dispenser. An existing product structured with castor wax and a silicone wax had a hardness by texture analyser of 0.0231 N/mm², a whiteness measurement after 1 hour of 23 and a whiteness measurement after 24 hours of 42. A competitor's product, believed also to have a wax structuring system had a hardness of 0.0318 N/mm², a whiteness measurement after 1

- 48 -

hour of 20, a whiteness measurement after 24 hours of 83, and a % transmittance of 0.004.

EXAMPLES

5

The examples set out in the Tables below were prepared using a number of materials for which the suppliers and proprietary names are given in the following list.

1. Rheoparl KL from Chiba Flour Milling Co.
- 10 2. GP-1 from Ajinomoto
3. Silicone wax from Dow Corning
4. Finsolv TN from Finetex
5. Parsol MCX from Givaudan-Roure
6. Sirius M70 from Dalton
- 15 7. Volatile cyclic silicone (cyclomethicone)
DC 245 from Dow Corning
8. Milled activated aluminium chlorhydrate from Summit
Water content 7.7%
9. MicroDry [™] aluminium chlorhydrate from Reheis
- 20 10. Al/Zr Tetrachlorohydrax glycine complex containing
about 10% water by weight (AZAG - 7167) from Summit
11. AZAG - 7167 with water content increased to 19% by
exposure in a humidity chamber at 85% RH
12. Milled activated aluminium chlorhydrate from Summit
25 (water content 17.2%)
13. Syncrowax ERLC, C₁₈₋₃₆ glycol esters (Croda)
14. Castorwax MP80, hydrogenated castor oil (CasChem)
15. Triacntenyl vinyl pyrrolidone copolymer, Antaron
WP-660 (ISP)
- 30 16. Paraffin wax, SP173P (Strahl and Pitsch)

- 49 -

17. Milled activated aluminium chlorohydrate from Guilini
18. AAACH Milled (super-active aluminium chlorohydrate)
19. Finsolv BOD (Finetex)
- 5 20. Finsolv SB (Finetex)
21. Finsolv TPP, mixture of C₁₂₋₁₅ alkyl benzoate / dipropylene glycol benzoate / PPG-15 stearyl ether benzoate (Finetex)
22. 1,1,5,5 -Tetraphenyl 1,3,3,5- tetramethylsiloxane
10 PDM 7040 (Gelest)
23. Octyldodecanol, Eutanol G (Henkel)
24. 12-Hydroxystearic acid (CasChem)
25. K7, trans-(1R,2R)-di-dodecanamido cyclohexane (of J3549)
- 15 26. K41, 2-dodecyl-N,N'-dibutylsuccinamide (prepared as described in WO 98/27955)
27. Waxenol 822 (Paroxite)
28. Masilwax 135, stearoxymethicone/dimethicone copolymer (BASF)
- 20 29. Aerosil 200 (Degussa)

The following general method of preparation was used for these examples. A solution of the thickening polymer and other structurants in the organic liquid(s) was made by
25 mixing these materials, heating and agitating the mixture at a temperature sufficiently high that the polymer and other structurants all dissolve. The mixture was then allowed to cool to 80-85 C before the aluminium antiperspirant active was added. The mixture was next allowed to cool to 5-30 C
30 above its gelling temperature (determined in a preliminary experiment) and poured into dispensing containers as

- 50 -

illustrated in Figs. 7 to 9 of the drawings. These were then left to cool to room temperature.

The procedure was varied slightly if GP-1 was used. This
5 was first dissolved in the hot liquid mixture after which
the thickening polymer and any other structurants were added
and dissolved. The general method of preparation then
continued as stated above with cooling to 80-85 C for the
addition of the antiperspirant active, further cooling to 5
10 to 30 C above the gelling temperature (determined
previously) and pouring into dispensing containers.

The process was varied slightly if silica was used. In an
initial step, the silica was mixed into the cold solvent
15 using a high shear mixer (Silverson). The procedure than
continued for introduction of the remaining constituents as
in the general method of preparation.

To raise the water content of AZAG-7167 from 10% as supplied
20 (material 10 above) to 19% as used in Example 8, (material
11 above) a quantity of the material was exposed to a
relative humidity of 85%, in a storage chamber maintained at
this humidity. The water content of the stored material was
determined analytically after 24 hours, and again after
25 exposure for a longer period, until the water content of 19%
was reached.

Determination of whiteness, transmittance and hardness was
done by the methods given earlier. All temperatures are in
30 degrees Celsius. Refractive indices were measured at
22.5 C.

- 51 -

Table 1

Ingredients	Percentages by Weight				
	Ex 1	Ex 2	Ex 3	Ex 4	Ex 5
Dextrin palmitate (1)	10	10	10	10	5
N-lauroyl-glutamic acid di-n-butylamide (2)	-	-	-	-	1
C12-15 Alkyl benzoate (4)	63.5	66	50.8	-	67.5
Mineral oil (6)	-	-	-	63.5	-
Cyclomethicone (7)	-	-	12.7	-	-
AACH (8)	26.5	24	26.5	26.5	26.5
Properties					
Refractive index of antiperspirant active	1.538	1.538	1.538	1.538	1.538
Refractive index of liquid	1.484	1.484	1.467	1.463	1.484
% transmittance	0.103	0.069	0.051	0.058	0.095
Hardness (N/mm ²)	0.039	0.030	0.031	0.015	0.017
Whiteness measurement (1 hr)	12	15	14	13	12
Whiteness measurement (24 hrs)	12	14	12	12	11

- 52 -

Table 2

Ingredients	Percentages by Weight				
	Ex 6	Ex 7	Ex 8	Ex 9	Ex 10 (comparative)
Dextrin palmitate (1)	5	10	10	10	10
Silicone wax AMS-C30 (3)	5	-	-	-	-
C12-15 Alkyl benzoate (4)	63.5	32.75	63.5	66	12.7
Octylmethoxy cinnamate (5)	-	31.75	-	-	-
Cyclomethicone (7)	-	-	-	-	50.8
AACH (8)	26.5	-	-	-	-
ACH (9)	-	-	-	24	-
AZAG 7167 (10)	-	26.5	-	-	26.5
Hydrated AZAG 7167 (11)	-	-	26.5	-	-
Properties					
Refractive index of antiperspirant active	1.538	1.562	1.528	1.524	1.562
Refractive index of liquid	1.484	1.513	1.484	1.484	1.417
% transmittance	0.052	0.023	0.035	0.048	0.005
Hardness (N/mm ²)	0.021	0.033	0.034	0.030	0.026
Whiteness measurement (1 hr)	14	17	14	16	13
Whiteness measurement (24 hrs)	12	15	14	13	11

- 53 -

The compositions of all these Examples were observed to break down when applied to the skin after extrusion from a dispensing container; this extrudate could be rubbed into skin easily.

5

When extruded from the container, the compositions of Examples 1 to 9 were observed to have a translucent appearance. The composition of Example 10 appeared white and opaque although the container construction was

10 identical.

- 54 -

Table 3

Alternative Structurants					
	Percentages by Weight				
Ingredients	Ex11	Ex12	Ex13	Ex14	Ex15
Dextrin Palmitate (1)	5	5			
N-Lauroyl-glutamic acid di-n-butylamide (2)	0.85			1	
Syncrowax ERLC (13)		3.75	2.5		
Castor wax (14)		1.25	7.5		
Triacontenyl vinyl pyrrolidone copolymer (15)				5	5
Paraffin wax (16)					5
C12-15 alkyl benzoate (4)	68.65	64.5	64.5	68.5	64.5
AACH A418 (8)					25.5
AACH A418 (12)	25.5	25.5	25.5	25.5	
Properties					
RI Active	1.512	1.512	1.512	1.512	1.538
RI Solvent	1.484	1.484	1.484	1.484	1.484
% Transmittance	0.14	0.13	0.16	0.11	0.05
Hardness (N/mm ²)	0.0159	0.0086	0.0388	0.0314	0.0122
White Deposits (1hr)	8	9	9	9	n/d
White Deposits (24hr)	7	8	9	9	n/d

- 55 -

Table 4

Alternative Structurants					
	Percentages by Weight				
Ingredients	Ex16	Ex17	Ex18	Ex19	Ex20
Dextrin Palmitate (1)	5	5	5		
12-Hydroxystearic acid (24)	4				
K7 (25)		1			
K41 (26)			1		
C12-15 alkyl benzoate (4)	65.5	68.5	68.5	64.5	64.5
AACH A418 (12)	25.5	25.5	25.5	25.5	25.5
Arachidyl Behenate (27)				2.5	
Silicone wax (28)					2.5
Castor wax (14)				7.5	7.5
Properties					
RI Active	1.512	1.512	1.512	1.512	1.512
RI Solvent	1.484	1.484	1.484	1.484	1.484
% Transmittance	0.10	0.21	0.16	0.35	0.40
Hardness (N/mm ²)	0.008	0.005	0.012	n/d	n/d
	5	8	8		
White Deposits (1hr)	n/d	n/d	n/d	n/d	n/d
White Deposits (24hr)	n/d	n/d	n/d	n/d	n/d

- 56 -

Table 5

Alternative Antiperspirant Actives		
	% by weight	
Ingredients	Ex21	Ex22
Dextrin Palmitate (1)	10	10
C12-15 alkyl benzoate (4)	64.5	63.5
Aloxicoll LR (17)	25.5	
AAACH milled (18)		26.5
Properties		
RI Active	1.538	1.526
RI Solvent	1.484	1.484
% Transmittance	0.07	0.04
Hardness (N/mm ²)	0.0375	n/d
White Deposits (1hr)	n/d	n/d
White Deposits (24hr)	n/d	n/d

Table 6

Alternative Emollient Oils						
	percentages by weight					
Ingredients	Ex23	Ex24	Ex25	Ex26	Ex27	Ex28
Dextrin Palmitate (1)	10	10	10	5	10	5
N-Lauroyl-glutamic acid di-n-butylamide (2)				1		1
C12-15 alkyl benzoate (4)					19.2	20.4
octyl dodecyl benzoate (19)	64.5					
isostearyl benzoate (20)		64.5				
benzoate mixture (21)			64.5			
Tetraphenyl tetra- methyilsiloxane (22)				53	44.8	47.6
Octyldodecanol (23)				15		
AACH A418 (8)	25.5	25.5		26		
AACH A418 (12)			25.5		26	
AZAG 7167 (10)						26
Properties						
RI Active	1.538	1.538	1.512	1.538	1.512	1.562
RI Solvent	1.4839	1.4860	1.492	1.533	1.534	1.534
% Transmittance	0.08	0.08	0.22	0.23	0.13	0.03
Hardness (N/mm ²)	0.0106	0.0159	0.0124	0.016	n/d	0.0173
White Deposits (1hr)	n/d	n/d	n/d	13	14	n/d
White Deposits (24hr)	n/d	n/d	n/d	11	14	n/d

- 58 -

Table 7

Inorganic Thickener					
	percentage by weight				
Ingredients	Ex29	Ex30	Ex31	Ex32	Ex33
Dextrin Palmitate (1)	5	5			
N-Lauroyl-glutamic acid di-n-butylamide (2)	0.85	0.85			
Syncrowax ERLC (13)			2.5	2.5	2.5
Castor wax (14)			7.5	7.5	7.5
Silica (29)	0.5	1.0	0.5	0.25	1.0
C12-15 alkyl benzoate (4)	68.5	67.5	64		
benzoate mixture (21)				64.25	63.5
AACH A418 (8)			25.5	25.5	25.5
AACH A418 (12)	25.5	25.5			
Properties					
RI Active	1.512	1.512	1.538	1.538	1.538
RI Solvent	1.484	1.484	1.484	1.492	1.492
% Transmittance	0.15	0.15	0.06	0.07	0.06
Hardness (N/mm ²)	0.0170	0.0278	0.0741	0.0287	0.0499
White Deposits (1hr)	n/d	n/d	n/d	n/d	n/d
White Deposits (24hr)	n/d	n/d	n/d	n/d	n/d

None of the silica containing formulations left any visible white deposits when applied to skin.

5

All of the formulations described in Examples 11 to 33 when extruded from the container through the narrow apertures were translucent to the eye of an experienced technician.

- 59 -

CLAIMS

1. An antiperspirant product comprising:
 - 5 (i) a dispensing container having at least one outlet aperture for delivery of container contents and having provision for a user to expel material from the container through the outlet aperture(s), and
 - 10 ii) within said container, a soft solid antiperspirant composition comprising a continuous phase of water-immiscible liquid, at least one structuring material which thickens said liquid and a particulate antiperspirant active in suspension in said liquid;
 - 15 wherein the refractive indices of said antiperspirant active and said liquid differ by not more than 0.10, and preferably by not more than 0.08.
2. A product according to claim 1 wherein the structuring
20 material is selected from the group consisting of organic polymer which is effective to increase the viscosity of the water-immiscible liquid, structurant which forms a network of fibres within the continuous phase,
25 waxes, other than fatty alcohols, which are solid at temperatures of 30 C and below, but melt below 95 C, mixtures thereof.
3. A product according to claim 2 wherein the structuring
30 material comprises 5 to 20% by weight of the composition, of an organic polymer which is effective

- 60 -

to increase the viscosity of the water-immiscible liquid.

4. A product according to claim 2 wherein the structuring material comprises :
- 5
- i) 1.5 to 15% by weight of the composition, of an organic polymer which is effective to increase the viscosity of the water-immiscible liquid;
- 10 ii) second structuring material selected from the group consisting of:
- 0.5 to 7% by weight of the composition of structurant which forms a network of fibres within the continuous phase,
- 15
- 3 to 10% by weight of the composition of waxes, other than fatty alcohols, which are solid at temperatures of 30°C and below, but melt below 95°C, and
- 20 mixtures thereof.
5. A product according to claim 4 wherein the second structuring material comprises 1 to 7% by weight of the composition of structurant which forms a network of fibres within the continuous phase.
- 25
6. A product according to claim 4 or claim 5 wherein the total amount of said organic polymer (i) and second structuring material (ii) is from 3% to 12% by weight of the composition.
- 30

- 61 -

7. A product according to claim 4 or claim 5 wherein the total amount of said organic polymer (i) and second structuring material (ii) is from 4% to 10% by weight of the composition.
- 5
8. A product according to claim 4 or claim 5 wherein the total amount of second structuring material is from 1% to 7% by weight of the composition.
- 10 9. A product according to any one of claims 2 to 8 wherein the organic polymeric thickener comprises from 2% to 7% by weight of the composition of a polysaccharide esterified with a monocarboxylic acid of 8 to 22 carbon atoms.
- 15
10. A product according to any one of claims 2 to 9 wherein organic polymeric thickener comprises from 3 to 12% by weight of the composition of a polymer selected from the group consisting of polyamides and hydrocarbon polymers.
- 20
11. A product according to claim 2 wherein the structuring material comprises from 1% to 6% by weight of the composition of gelating structurant which forms a network of fibres and/or from 3% to 8% by weight of the composition of said wax other than fatty alcohol.
- 25
12. A product according to claim 2 wherein the structuring material comprises from 4% to 8% of wax which is solid
- 30

- 62 -

at temperatures of 40°C and below, but melts below 90°C.

13. A product according to any one of the preceding claims
5 wherein the total amount of any fatty alcohol which is solid at 20°C is not more than 5%, preferably not more than 2% by weight of the composition.
14. A product according to any preceding claim comprising
10 from 0.1 to 2% by weight of an inorganic thickener.
15. A product according to any one of the preceding claims characterised in that the water-immiscible liquid contains a volatile silicone and optionally a non-
15 volatile silicone and/or a non-silicone hydrophobic organic liquid selected from hydrocarbons, hydrophobic aliphatic esters, aromatic esters and hydrophobic alcohols. A product according to any one of the preceding claims wherein the water-immiscible liquid
20 contains silicone oil in an amount which is from 5% to 20% by weight of the water-immiscible liquid.
16. A product according to any one of the preceding claims wherein the antiperspirant active comprises an
25 aluminium and/or zirconium halohydrate, an activated aluminium and/or zirconium halohydrate, or an aluminium and/or zirconium complex or an activated aluminium and/or zirconium complex.
- 30 17. A product according to claim 16 wherein the antiperspirant active comprises a halohydrate or

- 63 -

complex in which aluminium and zirconium are both present.

18. A product according to any one of the preceding claims
5 wherein the proportion of antiperspirant active is from 5 to 40% by weight of the composition.
19. A product according to any of claims 15 to 17 wherein
10 the antiperspirant active is in milled form.
20. A product according to any one of claims 15 to 19
wherein the antiperspirant active has a water content
of from 6 to 18% by weight.
- 15 21. A product according to claim 20 wherein the antiperspirant active has a water content of from 11 to 18% by weight.
22. A product according to any one of the preceding claims
20 wherein the refractive index of the water-immiscible liquid differs from that of the antiperspirant active by at least 0.01 but less than 0.06.
23. A product according to any one of the preceding claims
25 wherein the refractive index of the water-immiscible liquid is at least 1.45.
24. A product according to any one of the preceding claims
30 wherein said outlet aperture or each of said outlet apertures has an individual cross section whose smallest width is not more than 0.5cm.

- 64 -

25. A product according to any one of the preceding claims wherein the container has a plurality of said apertures for extrusion of the contents, each of which has a cross section whose maximum width is not more than 0.5cm.
26. A product according to any one of the preceding claims wherein said container comprises user-operable movable means to propel container contents towards and through said outlet aperture(s).
27. A product according to claim 23 wherein said means to propel container contents comprises a piston within said container and drive means to move said piston towards said outlet aperture(s).
28. A product according to claim 23 wherein said means to propel container contents comprises flexible container walls whereby the user can squeeze and compress said container.
29. A product according to any preceding claim wherein the outlet aperture in the dispensing container is sufficiently narrow that contents dispensed through the aperture is translucent.
30. A method of making a composition as defined in any one of the preceding claims, comprising:-
mixing the ingredients of the composition and,
before or after complete mixing, heating the
ingredients of the composition to a temperature at

- 65 -

which the structuring material is dissolved in the
water-immiscible liquid,
introducing the composition, at a temperature at which
it is mobile, into containers,

5 causing or allowing further cooling of the containers,
without further mixing of the composition, until the
temperature of the composition in the containers has
fallen below 30 C.

10 31. Topical application of a product according to any one
of claims 1 to 25 delivered through an aperture of up
to 5mm width.

Fig.1.

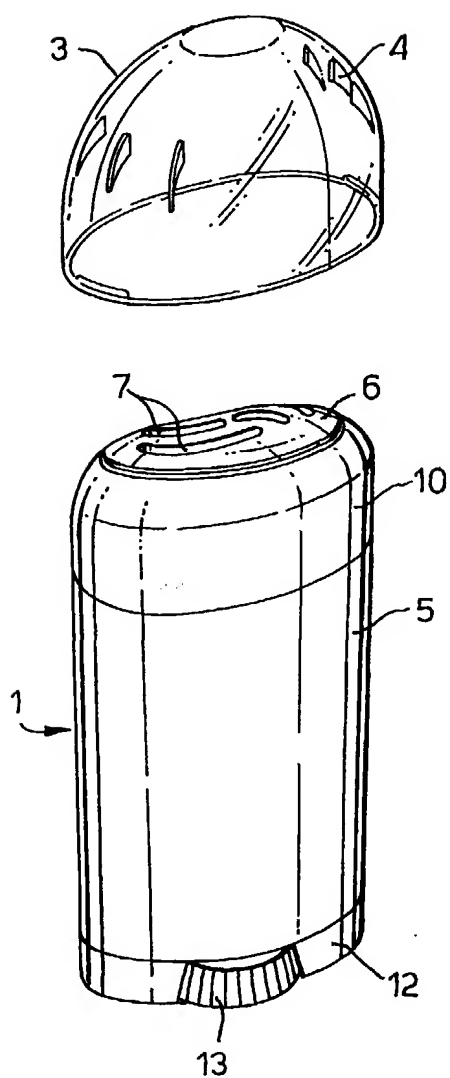
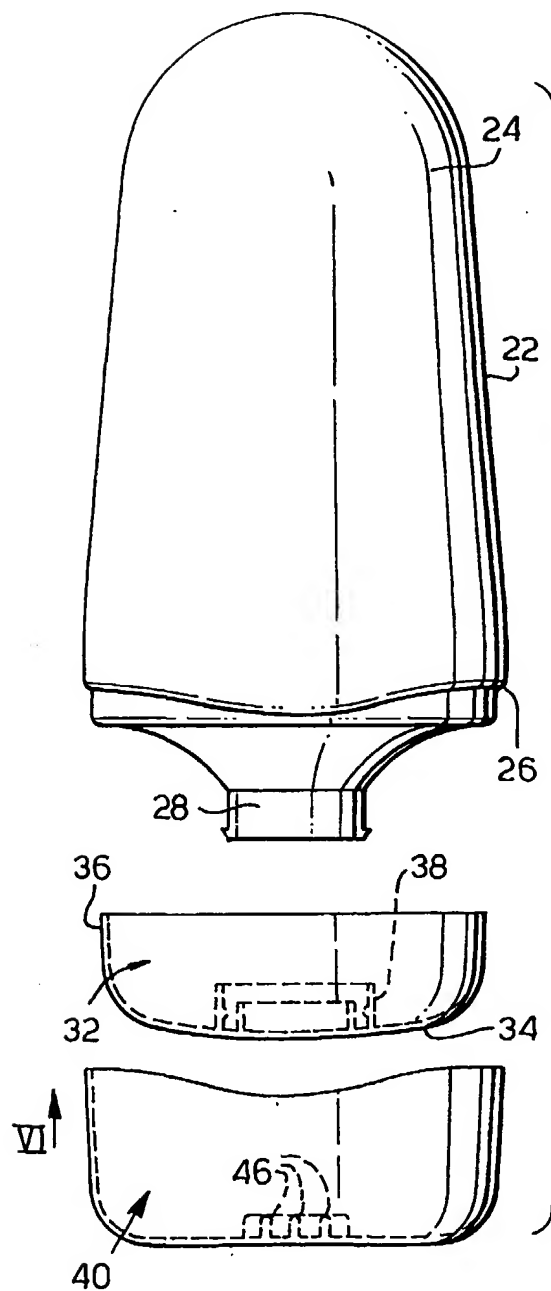


Fig.5.



2 / 3

Fig.2.

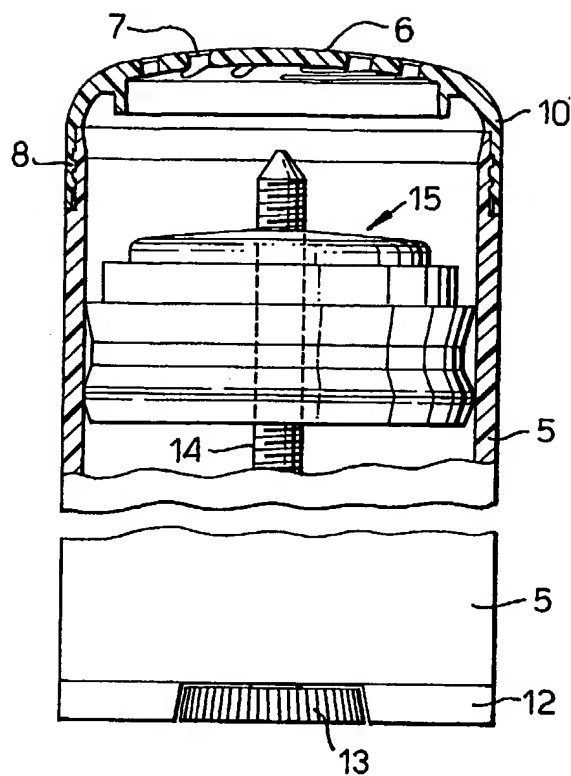


Fig.3.

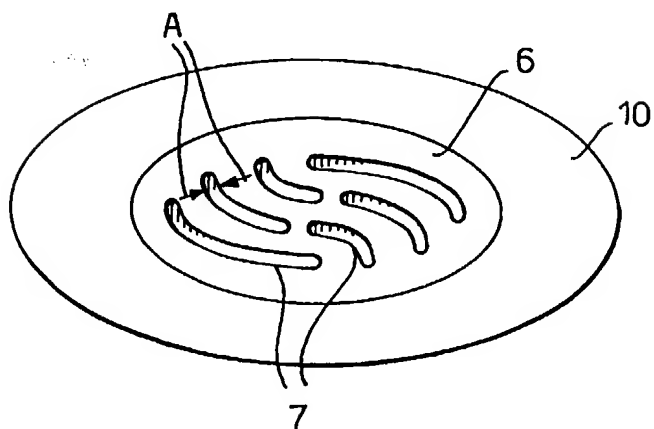
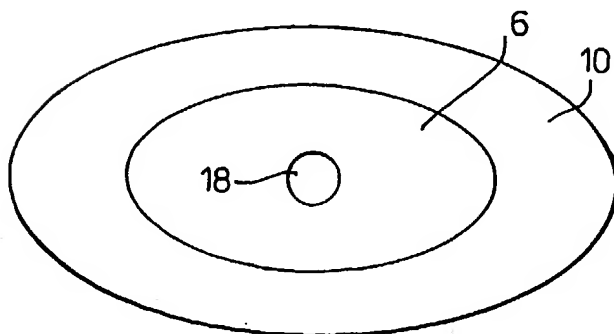


Fig.4.



3/3

Fig.6.

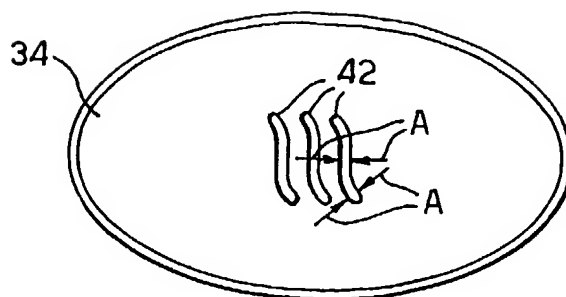


Fig.7.

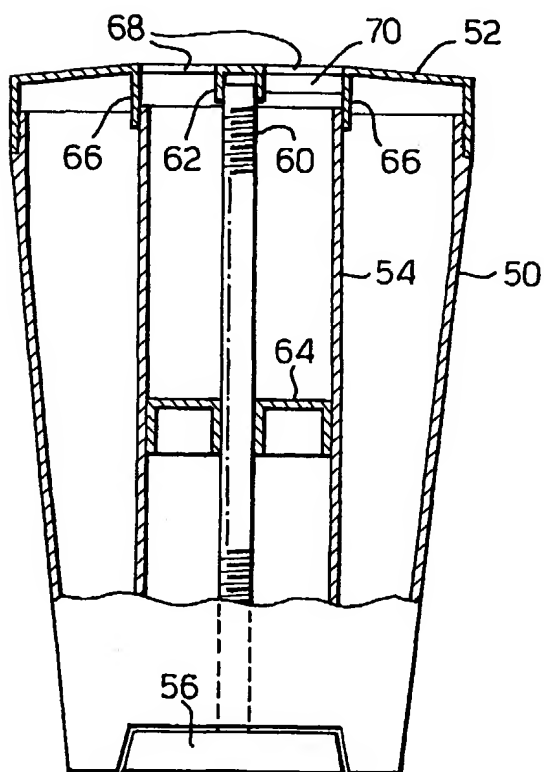


Fig.8.

